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IS 11255-6 (1999): Methods of Measurement of Emissions From Stationary Sources, Part 6: Ammonia [CHD 32: Environmental Protection and Waste Management]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
स्थिर स्रोतों से उत्सर्जन-मापन की पद्धतियाँ
भाग 6 अमोनिया

Indian Standard

**METHODS OF MEASUREMENT OF EMISSIONS
FROM STATIONARY SOURCES**

PART 6 AMMONIA

ICS 13.040.40

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Environment Protection Sectional Committee had been approved by the Chemical Division Council.

The undesired impacts of pollutants on flora and fauna is of high concern to scientists and technologists, in particular and to common people, in general. Ammonia is one of such pollutants, discharged in air mainly by fertilizer industry. Ammonia is known to be causing irritant to skin, eyes and respiratory tracts, which may become chronic with repeated exposure to its vapours. Keeping these in view, methods for measurement of ammonia has gained importance. This standard details two methods for determination of ammonia from stationary sources.

The composition of the technical committee responsible for preparation of this standard is given in Annex A.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF MEASUREMENT OF EMISSIONS FROM STATIONARY SOURCES

PART 6 AMMONIA

1 SCOPE

This standard prescribes two methods namely, Method A — Titration method and Method B — Nessler reagent method/Spectrophotometric method, for determination of ammonia from stationary sources.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
4167 : 1980	Glossary of terms relating to air pollution (<i>first revision</i>)
5182 (Part 5) : 1975	Methods for measurement of air pollution: Part 5 Sampling of gaseous pollutants

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 4167 shall apply.

4 METHOD A — TITRATION METHOD

4.1 Principle

Ammonia is collected in dilute sulphuric acid solution in two all-glass impingers to form ammonium sulphate. The sample solution is buffered at pH 9.5 with borate buffer and distilled into a solution of boric acid. The ammonia content in the sample is determined by titration of the distillate with a dilute standard sulphuric acid solution.

4.2 Range and Sensitivity

This method may be used to detect as little as 0.034 mg of ammonia in the sample. The lower detection limit is about 0.3 mg of ammonia in a 60 l gas sample, corresponding to a concentration of about 5 mg/Nm³. The upper detection limit may be extended by increasing the strength of the titrant acid solution.

4.3 Interferences

Ammonium salts will liberate ammonia during distillation to give high ammonia concentration. These can be removed by filtration of the gas sample before its passage into the impingers. Acidic and alkaline gases in the gas sample may interfere during titration. This is avoided through preliminary distillation step.

4.4 Apparatus

4.4.1 Probe

Chemical resistant glass tube, with an internal diameter of 5 to 6 mm with a heating system to prevent condensation and a filtering medium at the sample inlet end to remove particulate matter (end packed with glass or quartz wool).

4.4.2 Impinger

Three standard large impingers [see IS 5182 (Part 5)] connected in series and placed in an ice bath.

4.4.3 Pump

Leak-proof vacuum pump, capable of drawing upto 5 l/min air under the sampling conditions.

4.4.4 Valve

Needle valve or equivalent to adjust flow rate accurately in the range of 0.5 to 5 l/min.

4.4.5 Rotameter

Rotameter, orifice-meter or other suitable device to measure flow rate in the range of 0.5 to 5 l/min.

4.4.6 Dry Gas Meter

Sufficiently accurate to measure the sample volume within 1 percent, fitted with a thermometer and a pressure gauge/manometer to measure the metering temperature and pressure.

4.4.7 Glass Wash Bottle

Containing distilled water to rinse the impingers and connecting tubes.

4.4.8 Polyethylene Storage Bottles

With 100 ml capacity to store the impinger samples.

4.4.9 Distillation Apparatus

Borosilicate glass flask of 1 000 ml capacity attached to a vertical condenser so that the outlet tip may be submerged below the surface of the acid solution in the receiving flask.

4.4.10 pH Meter

4.4.11 Erlenmeyer Flask

500 ml capacity.

4.4.12 Burette

25 ml and 50 ml capacity with 0.1 ml sub division.

4.5 Reagents

4.5.1 Water

Deionized or distilled water, free of ammonia.

4.5.2 Absorbing Solution (0.1 N Sulphuric Acid)

Dilute 2.8 ml of concentrated sulphuric acid to 100 ml with distilled water.

4.5.3 Borate Buffer Solution

Dissolve 4.75 g of borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) in 500 ml of water. To this, add 88 ml of 0.1 N sodium hydroxide and dilute to 1 l.

4.5.4 Neutralization Agent

Sodium hydroxide, 1N.

4.5.5 Mixed Indicator Solution

Dissolve 200 mg of methyl red indicator in 100 ml of 95 percent ethyl alcohol or isopropyl alcohol. Dissolve 100 mg of methylene blue in 50 ml of 95 percent ethyl alcohol or isopropyl alcohol. Mix the two solutions. Prepare such mixed indicator solutions, once in a month, if needed.

4.5.6 Absorbent Solution

Dissolve 20 g of boric acid (H_3BO_3) in ammonia-free distilled water, add 10 ml of mixed indicator solution and dilute to 1 l. Prepare once in a month, if needed.

4.5.7 Standard Sulphuric Acid Titrant — 0.02 N.

Dilute 200 ml of 0.1 N sulphuric acid to 1 l. Standardize this by titrating against a known amount of sodium carbonate that has been incorporated in the boric acid solution, using the mixed indicator. One ml of acid solution is equivalent to 0.34 mg of ammonia.

4.6 Procedure

4.6.1 Preparation of Sampling Train

Pour 30 ml of 0.1 N sulphuric acid into each of the first two impingers. Leave the final impinger dry. Connect all the three impingers in series and place in the ice bath. Assemble the train as shown in Fig. 1. Check the sampling train for leakage at the sampling site by plugging the probe inlet and creating a vacuum

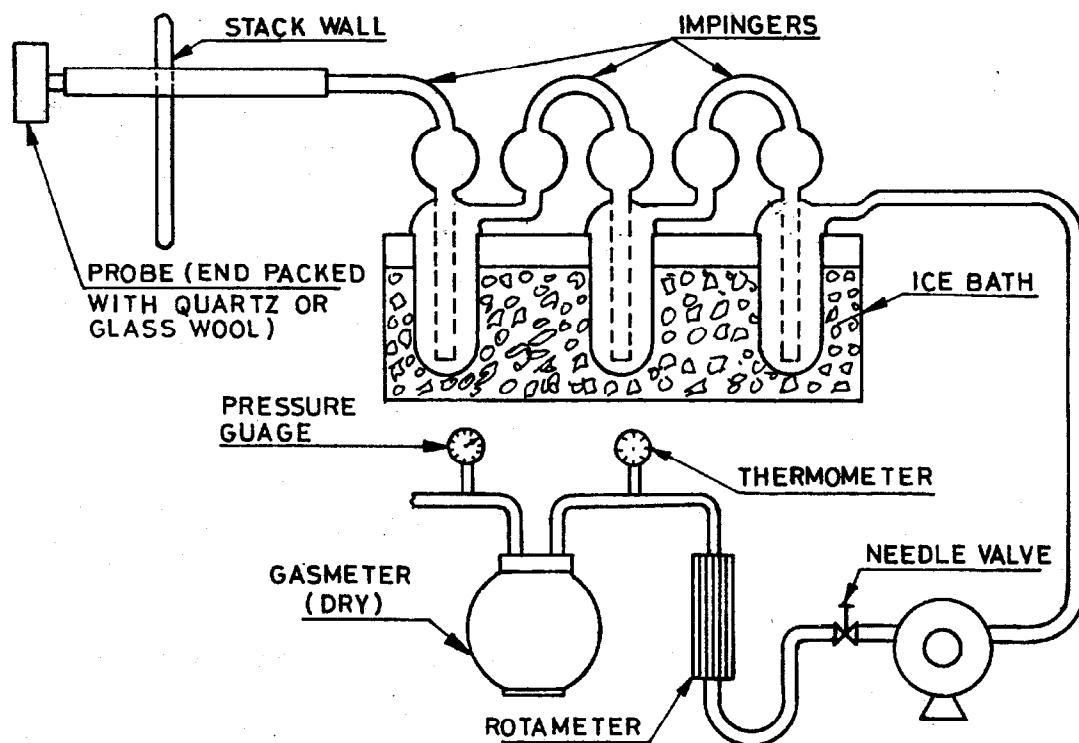


FIG. 1 AMMONIA SAMPLING TRAIN

corresponding to 250 mm mercury column. A leakage rate, within 1 percent of the sampling rate is acceptable. Add ice into the ice bath during the sampling to maintain the temperature of the gases leaving the impinger train at 20°C or less.

4.6.2 Sample Collection

Insert the sampling probe into the stack. Record the initial reading of the dry gas meter. Position the tip of the probe at the first sampling point within the stack. Start the pump and maintain the sample flow rate at about 2 l/min. Record the metering temperature and pressure between 2 to 3 min of sampling. Draw the sample for 5 min from the first point. Without stopping the pump, shift the tip of the probe to the second point. In the same manner, continue the sampling for a total period of 30 min, drawing the sample from 6 different points within the duct. Record the metering temperature and pressure at 5 min intervals. Maintain the sample flow rate at about 2 l/min by operating the valve. At the end of the sampling, turn off the pump and record the final reading of the gas meter. Remove the probe from the stack and disconnect it from the train. Preferably, sample shall be analyzed immediately. In case of delay, it shall be stored in a cool place.

4.6.3 Sample Recovery

Disconnect the impingers. Pour the contents of the first two impingers into a polyethylene shipment bottle. Rinse the two impingers and the connecting tube with ammonia-free distilled water and add the washings into the shipment bottle.

4.6.4 Ammonia Distillation

Transfer the contents of the shipment bottle quantitatively into a distillation flask and make the volume upto to about 400 ml with distilled water. Neutralize to approximately pH 7 with sodium hydroxide using a pH meter and make this volume upto about 500 ml with distilled water. Add 25 ml of borate buffer solution and adjust the pH to 9.5 with 6 N sodium hydroxide using a pH meter. Connect the distillation flask to the distillation apparatus. Pour 50 ml of absorbent solution (indicator boric acid solution) in the Erlenmeyer flask and place it below the condenser of distillation apparatus with the tip of the delivery tube below the surface of the absorbent solution. Distill at the rate of 6 to 10 ml/min and collect the distillate into the Erlenmeyer flask containing the indicator boric acid absorbent solution. Collect at least 200 ml distillate. Lower the collected distillate free of contact with delivery tube and continue distillation for 2 min to clean condenser and delivery tube.

4.6.5 Analysis

Titrate ammonia in distillate with standard 0.02 N sulphuric acid titrant until indicator turns to a pale lavender. Carry a blank through all steps and apply the necessary correction to the results.

4.6.6 Calibration

Use approved methods and equipments to calibrate dry gas meter and rotameter. Standardize the 0.02 N sulphuric acid solution by titrating against a known amount of sodium carbonate in the boric acid absorbent indicator solution. Carry blank run with the sampling reagent (0.1 N sulphuric acid), consisting of distillation and titration steps.

4.7 Calculations

4.7.1 Dry Gas Volume

Correct the sample volume measured by the dry gas meter to normal conditions (298°K and 760 mm Hg) on dry basis by using the following equation:

$$V_n = V_m = \frac{298}{273 + t_m} \times \frac{P_a + P_m - P_v}{760}$$

where

V_n = volume in m^3 of dry gas sample under normal conditions;

V_m = volume in m^3 of dry sample measured in the gas meter under meter conditions;

t_m = average temperature in °C of gas sample through dry gas meter;

P_a = atmospheric pressure in mm Hg during sampling period;

P_m = average gauge pressure in mm Hg of gas sample through dry gas meter; and

P_v = saturated vapour pressure of steam at t_m .

4.7.2 Ammonia Concentration

Calculate the concentration of ammonia using the following equation:

$$C = 0.34 \times \frac{(V - V_b) \times f}{V_n}$$

where

C = concentration in mg/Nm^3 of ammonia in gas sample;

0.34 = mg of ammonia equivalent to 1 ml of 0.02 N sulphuric acid;

V = volume in ml of 0.02 N sulphuric acid used for the sample;

V_b = volume in ml of 0.02 N sulphuric acid used for the blank;

f = factor of 0.02 N sulphuric acid ; and

V_n = volume of dry gas sample, m^3 .

4.7.3 Ammonia Emission

Calculate the emission of ammonia from the stack as follows:

$$E = 10^{-3} C \times O_n$$

where

E = ammonia emission rate, g/h;

C = concentration of ammonia in the exhaust gas, mg/Nm³ (dry); and

O_n = exhaust gas flow rate, Nm³/h (dry).

4.8 Precision and Accuracy

The precision and accuracy of the procedure involving sampling, distillation and analysis have not been reported. Preliminary studies have indicated that the accuracy of the procedure is ± 5 percent at 100 mg/Nm³ and ± 10 percent at 20 mg/Nm³.

5 METHOD B — NESSLER REAGENT METHOD/SPECTROPHOTOMETRIC METHOD**5.1 Principle**

Ammonia is collected in a dilute sulphuric acid solution in a midjet impinger to form ammonium sulphate. The solution is reacted with Nessler reagent to produce a yellow-brown complex. The ammonia concentration is determined by reading the absorption of the yellow-brown solution at 440 nm and comparing it with a standard curve. Absorption peak may shift with concentration.

This method is sensitive, but ammonium salts can interfere. This method does not distinguish between free and combined ammonia.

5.2 Range and Sensitivity

The nessler reagent is sensitive to as little as 0.002 mg of ammonia. The range of application of this method is 0.1 mg to 0.80 mg of ammonia in a 10 l air sample.

5.3 Interferences

Ammonium salts will react with Nessler reagent to give a false high reading. These can be removed by filtration of the air before its passing into the absorption tube. Other interferences are not known.

5.4 Apparatus**5.4.1 Battery Operated Personal Air Sampling Pump**

Capable of drawing 1 l of air per minute through 10 ml of absorbing solution.

5.4.2 Impingers and Standard Midjet Impinger

All glass, calibrated.

5.4.3 Spectrophotometer**5.4.4 Spectrophotometer Cells**

1 cm.

5.4.5 Volumetric Flasks

50 ml, 100 ml, 200 ml, 1 000 ml glass stoppered.

5.4.6 Pipette

0.5 ml, 1.0 ml, 5 ml, 10 ml and 20 ml.

5.4.7 Graduated Cylinders

25 ml, 50 ml and 100 ml.

5.5 Reagents**5.5.1 Water**

Double distilled water, free of ammonia.

5.5.2 Mercuric Chloride**5.5.3 Potassium Hydroxide****5.5.4 Potassium Iodide****5.5.5 Ammonium Sulphate****5.5.6 Nessler Reagent**

Dissolve 35 g mercuric chloride in 500 ml of hot water. Filter and allow to cool. Dissolve 62.5 g of potassium iodide in 260 ml of cold water. Gradually, add the mercuric chloride solution to 250 ml of the iodide solution until a slight permanent red precipitate is formed. Dissolve the precipitate with the remaining iodide solution and again add mercuric chloride slowly until a red precipitate remains.

Dissolve 150 g of potassium hydroxide in 250 ml of distilled water. Add this solution to the potassium iodide-mercuric chloride solution and make up to 1 l with distilled water. Stir thoroughly and allow to stand a day or so, and decant the clear liquid.

NOTE — The Nessler reagent shall be handled with caution because of its toxicity and corrosive properties.

5.5.7 Standard Ammonium Sulphate Solution

Dissolve 77.6 mg of ammonium sulphate (desiccated overnight) in distilled water and make up to 1 l. One ml of this solution contains 20 µg of ammonia. Discard solution after a week.

5.5.8 Absorbing Solution

Dilute 2.8 ml of concentrated sulphuric acid (18 M) to 1 l with distilled water to form 0.1 N sulphuric acid.

5.6 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean that do not contain impurities which affect the results of analysis.

5.7 Procedure**5.7.1 Sampling**

Add 10 ml of absorbing solution to each midjet impinger including one for the blank. Attach the

bubbler to a personal air sampling pump and draw air through the bubbler at a rate of 1 l/min for 10 to 15 min. Record the volume of air sampled.

5.7.2 Colour Development

Dilute the sample to 50 ml with distilled water in a volumetric flask and shake well. Take 1 ml of this solution and make up to 50 ml with distilled water in another volumetric flask. Shake, add 2 ml of Nessler reagent to the latter flask and determine absorbance after 10 min at 440 nm in a spectrophotometer using a 1 cm cell. Treat the blank in the same manner as the sample.

5.8 Calibration and Standardization

Dilute samples of 5 ml, 10 ml, 20 ml, 30 ml and 40 ml

of the standard ammonium sulphate solution and process in the same manner as described in 5.7.2. Determine light absorbance in a spectrophotometer at 440 nm. Prepare a standard curve of absorbance versus mg of ammonia.

5.9 Calculation

$$\text{Ammonia, mg/Nm}^3 = \frac{M}{V}$$

where

M = mass in μg of ammonia from the standard curve minus blank value, and

V = volume in litre of air corrected to 25°C and 760 mm Hg.

ANNEX A
(Foreword)
COMMITTEE COMPOSITION

Air Environment Protection Sectional Committee, CHD 32

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<i>Members</i>	
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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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